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Functional Nanostructures for Induction Heating: A Review of Literature and Recommendations for Research

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Functional Nanostructures for Induction Heating: A Review of Literature and Recommendations for Research

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Abstract

This report presents the concept for a multidisciplinary research program aimed at establishing the science base for the design and synthesis of magnetic nanoparticles for hysteresis heating, with potential applications ranging from novel composites processing techniques to alternative cancer treatments. Magnetic materials are used in a wide range of applications and designed for maximum efficiency or minimized hysteresis loss. The uniqueness of this project is that, while considerable work has been aimed at reducing hysteresis losses, the converse effect (i.e., increasing hysteresis losses and therefore heat generation) has not been fully studied. Hysteresis-based heating has several advantages over conventional heating techniques, including the fact that it is a very rapid and noncontact process. In addition, the Curie temperature of magnetic materials can also be used as a means of "smart" thermal control. Exploratory basic research through ongoing programs at the U.S. Army Research Laboratory (ARL) has established the feasibility of Curie temperature control and demonstrated the effects of particle size, frequency, and stoichiometry on hysteresis losses. The following scientific barriers are addressed in this report: magnetization dynamics in high-frequency magnetic fields, the effects of magnetic phase transition on hysteresis heating, and the dimensional dependence of Curie temperature in nanoparticles. If carried out, the research program outlined in this report would establish the science base for the design and synthesis of nanoparticles for hysteresis heating applications.

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1. Introduction

The goal of this report is to outline the research requirements to establish the science base for the design and synthesis of nanoparticles for hysteresis heating, with potential applications ranging from novel polymer processing techniques to alternative cancer treatments. This report outlines a multidisciplinary research approach encompassing physics, material science, and biology research areas. The proposed research focuses on maximizing hysteresis losses in magnetic particle systems, while research to date has focused on minimizing hysteresis losses. As shown in Figure 1, a multidisciplinary research approach would enable advances in the basic science of hysteresis heating in magnetic nanoparticles and subsequent optimization of these nanoparticles for potential use in biological and polymer processing applications.

Interest in the study of nanostructured materials has increased rapidly during the past several years, stimulated by recent advances in materials synthesis and characterization techniques. These materials are of great scientific interest in developing a better understanding of magnetic phenomena. Their unique structure offers a fertile ground to study size, surface/interface, and intra- and interparticle interaction effects [1–3]. These materials are being used in a wide range of applications, including magnetic tapes, ferrofluids, catalysts, medical diagnostics, drug delivery systems, and pigments in paints and ceramics [1, 2, 4–6]. As never before, magnetic materials are the key to the future of the storage industry [7–13].

Research on fine magnetic particles started in the late 1940s [14, 15] and peaked in the 1950s. One of the milestones of that development was the emergence of domain theory [16], which led to the concept of single domain particles [17] and magnetic anisotropy contributions, giving rise to permanent magnet behavior [18] and superparamagnetism [19]. Stimulated by the potential application of fine magnetic particles in magnetic recording media [20, 21], most recent studies have concentrated on magnetization and coercivity [1, 2], magnetic reversal mechanism [22–25], and superparamagnetic relaxation [26, 27]. The dynamics of fine magnetic particles, including phenomena such as domain wall resonance and spin resonance, have rarely been

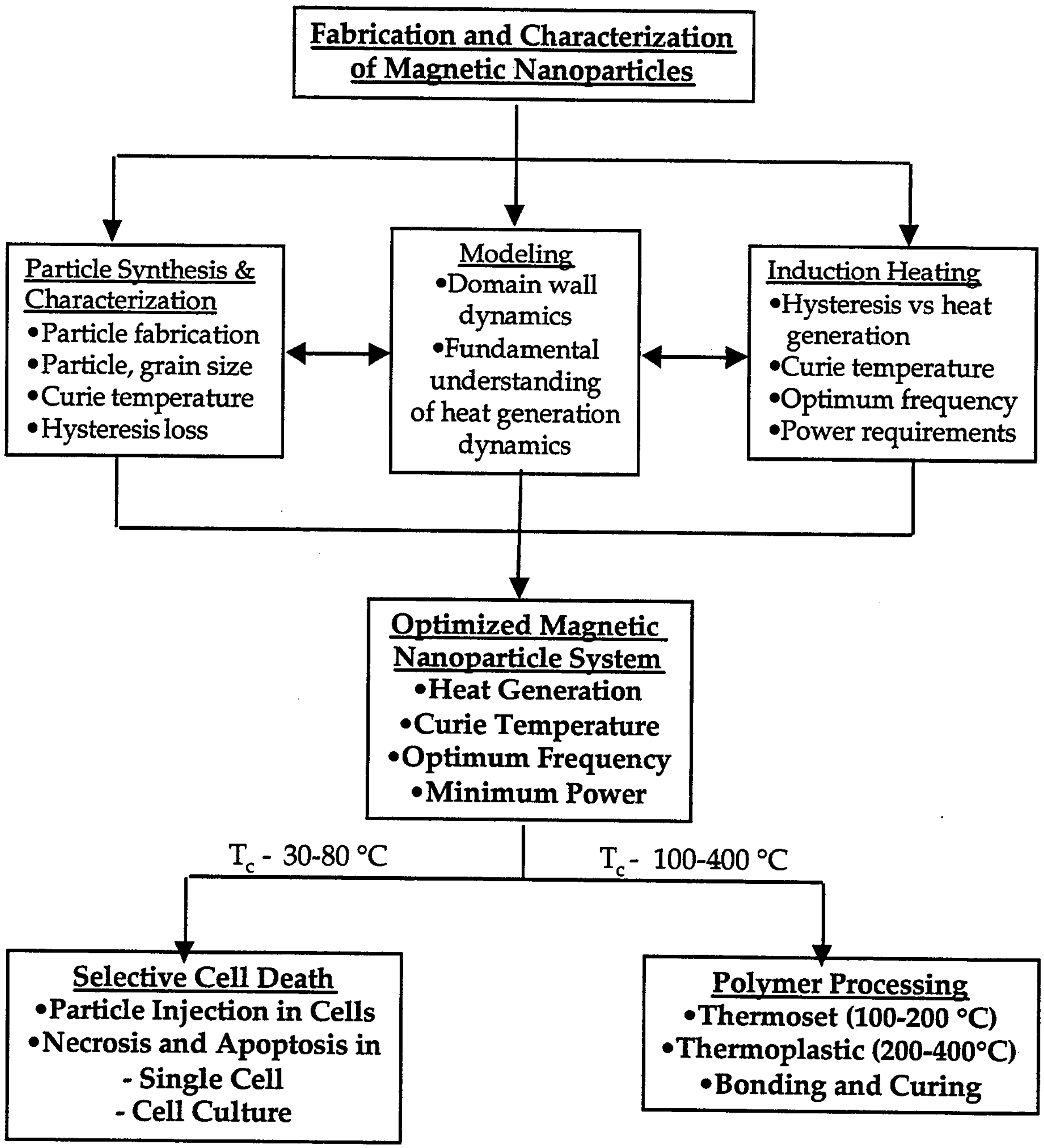

Figure 1. Multidisciplinary Research Effort in Hysteresis Heating of Magnetic Nanoparticles for Biological and Polymer Processing Applications.

studied, except in the superparamagnetic region. These phenomena are directly related to the conversion of energy absorbed from external magnetic fields to heat.

In most applications of magnetic nanoparticles, heat generation is not desirable. However, induction-based hysteresis heating has several features that can be exploited for efficient and economical heating. Two applications proposed as part of this effort use hysteresis heating of particles. The first involves induction heating for processing of advanced polymers; the second is targeted at the use of magnetic nanoparticles for selective heating and destruction of cells. The proposed effort addresses the scientific barriers to magnetic nanoparticle design for controlled heating.

Exploratory basic research at the U.S. Army Research Laboratory (ARL) at Aberdeen Proving Ground (APG) and the University of Delaware has established the feasibility of hysteresis heating for polymer processing [28–31], and an invention has been disclosed on this technology [32]. Existing processing techniques for polymers have time and cost limitations that can be overcome by hysteresis heating. In the repair of high-performance polymer composites where the thermal requirements are stringent, self-controlled heating based on the Curie temperature of the material is ideal. In addition, by using nanoparticles as additive fillers, one can easily adapt existing processing techniques for hysteresis-heating-based processing of polymers. New research is required to quantify the heat-generation capabilities of a variety of magnetic nanoparticle systems and the use of Curie temperature as a built-in "smart" thermal control system.

Hyperthermia in the treatment of cancer is well established in terms of the required temperatures and dwell cycles for cellular thermal shock, thermotolerance buildup from heat shock proteins, apoptosis, and rapid thermoablation [33–39]. The most clinically effective and desirable means of imposing thermal energy for hyperthermia is to inject a susceptor material directly into the target cells. This allows localization of thermal energy into the target cells alone. In order to be acceptable for hyperthermia treatment for humans, the susceptor material must be injectable into the cellular structure, must remain localized to the target cells for

subsequent treatment, and must not cause clinical complications. In order to be effective, the susceptor material, or ferrofluid, must be energetically able to remain in suspension, must be capable of rapid heating to the correct "process" temperature to minimize electromagnetic radiation exposure time for the patient, must have a Curie temperature that provides for precise control of temperature, and must have sufficient hysteretic properties such that the volumetric percentage of loading of particles is minimized. Most research to date has focused on the former "acceptibility" issues and not on the latter "effectiveness" issues. The proposed work would focus on the latter enabling issues for cancer treatment.

Magnetic materials have been considered for a number of therapeutic applications, including contrast imaging [40], magnetic guidance of drugs or radionucleides to selected target sites [41], and hyperthermic destruction of cells using inductive heating methods [42–44]. The latter application represents an important advance in cancer therapy and is the focus of this report. Two major obstacles to the implementation of such an approach can be imagined: (1) selective targeting of particles to specific cells and (2) optimization of particle and heating parameters to destroy those cells with minimal collateral damage. In addition, there are numerous other considerations, such as toxicity, metabolic clearance of particles, intracellular vs. extracellular efficacy, and precise control of heat production in the target area, vis-à-vis blood flow patterns, tissue density, etc. While many of these issues have been considered [42, 44–46], there has been little systematic investigation of nanoparticle properties in a reproducible bioassay. Thus, one objective of any new research thrust should be to develop such bioassays, based on nanoparticle-induced hyperthermic cell death.

Used in a wide range of applications, magnetic materials are designed for maximum efficiency or minimized power loss. One mechanism of power loss in these materials is magnetic hysteresis loss, which results in heating of the material. While considerable work has been aimed at reducing hysteresis losses, the converse effect—i.e., increasing hysteresis losses and therefore heat generation—has not been fully studied.

Hysteresis-based heating has several inherent advantages over other heating techniques.

- Energy input is noncontact.
- Heating can be very rapid (~50 °C/s).
- Heating can be self-controlling based on the Curie temperature of the material.

In order to fully exploit the potential of hysteresis heating, work is necessary to understand the relationships between hysteresis heating and material parameters, such as composition and particle size. Nanoscale particles allow us the flexibility of (1) using size-property relationships to maximize hysteresis losses, (2) minimizing eddy current losses such that Curie temperature can be for smart temperature control, and (3) maximizing particle-polymer interphase to optimize thermal and mechanical properties.

The following outlines a three-step research effort on the design and application of hysteresis heating of magnetic nanoparticles.

(1) Establish the science base to understand the hysteresis behavior of magnetic nanoparticles as a function of particle parameters, magnetic field, and frequency.

(2) Develop materials by design based on the physics of magnetic particles. With the science base established, given the heating requirements for specific applications, one can easily determine the specific material parameters to be used; this will involve fabricating of nanoparticles of a variety of compositions and particle sizes, characterizing of the magnetic and heating properties, and theoretical modeling to optimize particle preparation.

(3) Identify and demonstrate two potentially high-gain applications using the following nanoparticles:

- Curie temperature control-based polymer processing, and
- selective cell death—necrosis and apoptosis.

2. Scientific Barriers in Particle Design

2.1 Issues. For these objectives to be met, several basic issues in the physics of magnetic particles must be addressed.

- Development of reliable and controllable methods to synthesize nanoparticles.

- Development of an understanding of the magnetization dynamics in high-frequency magnetic fields, with the ultimate goal being to maximize the energy loss, which is minimized in traditional applications. These high-frequency behaviors, which have rarely been studied, are of great scientific interest, relating magnetic tunneling, domain wall damping, and domain wall resonance phenomena.

- Investigation of the effects of magnetic phase transition on hysteresis heating (energy loss); the magnetic transition of a nanoparticle encompasses ferromagnetic, superparamagnetic, and paramagnetic phases.

- Application-based identification of particle composition, size, microstructure, induction power, and frequency for maximum heat generation or power loss at a specified Curie temperature. The Curie temperature is chosen based on the applications; 100–400 °C for polymer processing and 30–80 °C for cell death studies.

2.2 Particle Synthesis. There are numerous techniques for preparing fine particles [1, 2]. These include electrodeposition [47], reducing transition metal ions by $NaBH_4$ or KBH_4 [48–51], sol-gel processes [52], spark erosion [53–55], aerosol pyrolysis [56], gas evaporation [20, 57], sputtering [26, 58], gas and water atomization [59], reverse micelle techniques [60–63], and mechanical ball milling (MB) [64–66]. MB, unlike many of the aforementioned methods,

produces its nanostructures not by cluster assembly but by the structure decomposition of coarse-grained structures as the result of severe plastic deformation in a cyclic process. This has become a popular method to make nanocrystalline materials because of its simplicity, the relatively low cost of the equipment needed, and the applicability of the method to essentially all classes of materials. Another major advantage is the potential for easily scaling up to tonnage quantities of materials for various applications.

It is necessary to first distinguish between nanoparticles and nanograin/nanocrystalline particles. The former refers to particles of nanometer size that generally consist of a single grain, and the latter to particles consisting of many grains of nanometer size. The nanograin particle may not be nanometer in size. For brittle materials, such as oxides, nitrides, carbides, and rare earth-transition metal intermetallic compounds, MB can indeed produce nanoparticles [67], whereas for ductile materials, straight MB will produce only nanograin particles because of the excessive cold welding. The nanoparticles can, however, be fabricated either using various surfactants (hexane) [68–70] or milling the material in an oxygen or hydrogen atmosphere and deoxidizing/dehydriding the final powders [59]. Both nanoparticles and nanograin particles are relevant to this research, as discussed in the following paragraphs.

A detailed microscopic model of the development of a nanocrystalline structure by MB is still lacking, although there is some agreement on the phenomenological model, which comprises three stages [64, 65].

(1) Deformation Stage—the deformation is localized in shear bands containing a high dislocation density.

(2) Nanograin Formation—dislocations annihilate and recombine to form nanograins with small-angle grain boundaries. Further milling extends this structure throughout the sample.

(3) Nanograin Randomization—the orientation of the grains becomes random, probably through grain boundaries by sliding or rotating.

These deformation processes are important for fundamental studies of extreme mechanical deformation and for developing a nanostructured state of matter with particular physical and chemical properties. The mechanisms of dislocation vs. grain boundary deformation can be distinguished by studying the atomic-level lattice strain (ε) and the stored enthalpy (ΔH) as a function of reciprocal grain size. At stage 1, both lattice strain and enthalpy depend weakly on grain size. A large jump in the values of ε and ΔH signifies the transition into stages 2 and 3. Finally, both ε and ΔH reach a maximum value before decreasing. The causes of such maxima are still under debate [66, 71–74]. It is important to identify the microstructure in which optimal magnetic properties can be realized.

Magnetic particles can be fabricated using a shake mill and a computer-controlled attrition mill. The latter has a variable rotor speed of up to 1,500 rpm, with a capacity up to 500 cm^3. Particles can be made in vacuum, gas atmosphere, or solution. For alloys, small particles are made either through mechanical alloying (mixing powders) or milling down the alloys, which are made using arc melting or vacuum-induction furnaces. Magnetic oxide particles, which may be more friendly to biological cells, can also be made using powders, which are readily available. To achieve the nanoparticle size for ductile materials, two methods will be used. Materials can be ball-milled in an oxygen or hydrogen environment. The interstitial oxygen/hydrogen or oxides/hydrides tend to make materials brittle. The final nanoparticles will be treated in a high vacuum to remove/disassociate the oxygen or hydrogen. An alternative way to fabricate nanoparticles is to add surfactants, usually a hexane solution, during the ball milling. For example, extremely soft aluminum and magnesium powders can be milled to form small alloy particles in the presence of sodium-1, 2 bis (dedecyl carbonyl) ethane-1-sulfonate or lithium-1,2 bis dodecyloxycarbonyl sulfasuccinate [68], and $(Co\text{-}Fe)_{75}Si_{15}B_{10}$ soft magnets can be milled down to about 100 nm in diameter in the presence of sodium-1,2 bis (dedecyl carbonyl) ethane-1-sulfonate or ammonium dihexadecyl dimethylacetate.

Particle size and grain size can be characterized using x-ray, electron microscopy, and particle analyzers (GALAI CIS100 and Brookhaven Instrument B1-XDC). The GALAI CIS100 analyzer measures particles from 0.5 to 600 μm in size by measuring the rotating laser time of shadowing, with additional particle shape analysis using a rapid video microscope picture analyzer. The Brookhaven analyzer uses centrifugal analysis with an x-ray detector and is capable of measuring particles from 10 nm to 10 μm in size. The stored enthalpy will be measured using differential scanning calorimetry (DSC) by integrating the exothermal signals. The structure will be carefully characterized and optimized to produce maximum heat. Such optimization can be achieved through adjusting rotor speed and adding appropriate surfactants.

A common problem encountered with milling fine particles is the potential for significant contamination from the milling media (balls and vial) or atmosphere. Such contamination can be significantly reduced using a low-energy mill [66], which can be achieved in our attrition mill by reducing the rotor speed. It has recently been shown [73] that the minimum nanocrystalline grain size for a number of elements milled in a low-energy mill is comparable to that from a high-energy mill [71, 74, 75]. Surfactants may also be used to minimize contamination. For example, boric acid and borox were quite effective in reducing iron contamination [76]. Our attrition mill is also capable of fabricating materials in a vacuum environment, thus minimizing atmospheric contamination.

2.3 Dynamics of Magnetic Domain Wall Motion. Many of the specific applications of magnetic materials depend on their behavior at high frequencies. When subjected to an alternating field, magnetic permeability shows dependence on several magnetization mechanisms. As the field frequency increases, magnetic moments are unable to follow the applied field because of "microeddy currents" generated by the ac field near the domain walls. Consequently, there is a lag between the applied field and the magnetization of the material, resulting in hysteresis-type behavior. The permeability becomes a complex number, and the imaginary part of the permeability corresponds to the energy dissipation. This energy dissipation appears in the form of heat and is commonly called hysteresis heating. In all nonheating

applications, hysteresis heating should be minimized; however, the proposed applications require the converse (maximum hysteresis heating and desired Curie temperature T_c).

The frequency spectrum for each magnetization mechanism is different, since each has a different time constant. In bulk materials, such as ferrites, the low-frequency (MHz) peak is associated with domain wall dynamics and the high-frequency peak, usually in the GHz range, with spin resonance. There are very few studies on the dynamics of magnetic particles, and the emphasis was exclusively on quantum tunneling behavior, which appear at sub-Kelvin temperature and low frequency (<1 MHz) [77, 78]. The study of particle dynamics will not only lead to a new understanding of their behavior in ac fields, but also significantly impact both heating and nonheating applications, as more materials based on particles are used.

Consider a 180° domain wall, as shown in Figure 2; an external magnetic field $H(x,z)e^{i\omega t}$ exerts a pressure on and bows the Blöch wall, displacing it a distance h(z) in the x-direction. These displacements approximately obey the equation [79, 80]

$$\gamma\frac{d^2h}{dz^2}-m\frac{d^2h}{dt^2}-\beta\frac{dh}{dt}-\alpha h=-2M_sH(h), \tag{1}$$

where all the coefficients are defined per unit area. The first term represents the wall energy, and γ is the wall surface tension. The second term is the wall inertia, and m is the effective wall mass. The third term is the relaxation damping term opposing wall propagation, and the last term is associated with wall pinning due to defects, expressed as a restoring force. The equation resembles a harmonic oscillator and has no analytical solution. Many assumptions have been made in order to solve the equation—e.g., either rigid or flexible plane walls (no bowing) were used in the Polivanov model [81] and the Pry and Bean model [82], respectively. In the models where wall bowing is considered, a linear response approximation was always used [79] (either H is not a function of h or only a linear term is considered).

Most recently, in an attempt to explain results on the frequency response of iron-cobalt soft magnetic materials at high temperatures [83], Chui reconsidered the problem [84]. Consider the situation shown in Figure 2; in addition to equation (1), the equation for H was also derived as follows:

$$\frac{\partial H}{\partial t}=\frac{c}{4\pi\sigma}\left\{\frac{\partial^2 H}{\partial z^2}+\left[1+\left(\frac{\partial h}{\partial z}\right)^2\right]\frac{\partial^2 H}{\partial x^2}-2\frac{\partial h}{\partial z}\frac{\partial^2 H}{\partial x\partial z}+\left[\left(\frac{c^2}{4\pi\sigma}\right)^{-1}\frac{\partial h}{\partial t}-\frac{\partial^2 h}{\partial z^2}\right]\frac{\partial H}{\partial x}\right\}, \quad (2)$$

where σ is electric conductivity. To avoid any simplifying assumptions, a numerical method was used to solve the equations. Similarly, core losses, which correspond to heat generation, can also be calculated by integrating $jE = \sigma E^2$. The electric field E can be calculated using $\nabla\times E = 1/c(\partial B/\partial t) = M_s/c(\partial h/\partial t)$. The first-order approximation, in the case shown in Figure 2, leads to $\nabla\times E \approx 2E$, and core loss will be proportional to the domain wall speed $(\partial h/\partial t)^2$. However, a detailed solution should be obtained by solving equations (1) and (2) self-consistently.

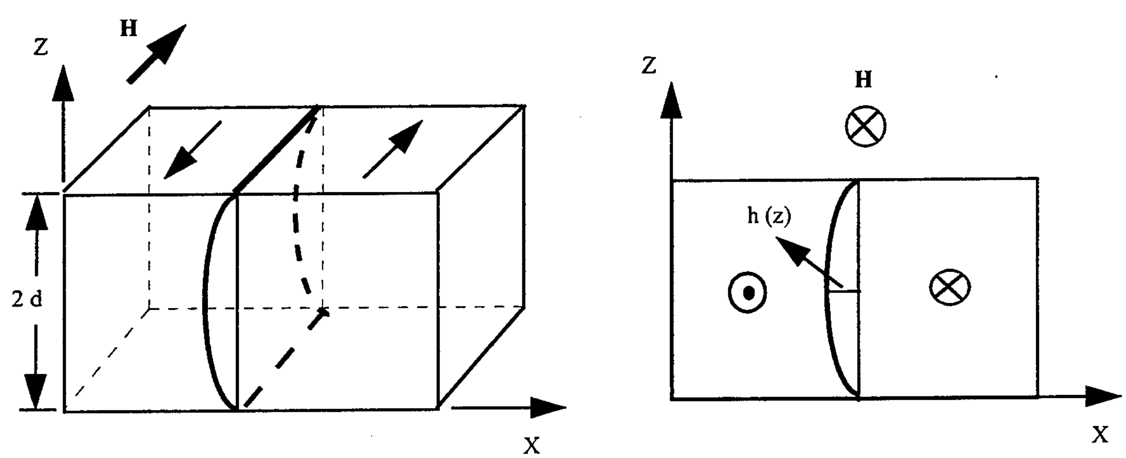

Figure 2. Schematic of Domain Wall Motion Due to External Field.

With some modifications, the previous method can be applied to magnetic particles. Particularly, anisotropy and dipolar energy, which scale with particle size, have to be considered.

The interplay between exchange interaction, anisotropy, and dipolar interaction leads to the formation of edge domains. The formation of edge domains and magnetization reversal mechanisms have recently been studied theoretically with the Monte-Carlo simulation by Chui using $4 \times 4 \times 12$ "block spins" in nanosize thin films [85], particles, and nanowires [86]. The finite element block spin, in which all atomic spins are aligned, is chosen so that the detail of domain formation can still be clearly seen, and the computing power (time) is minimized. Consequently, a relatively large sample can be simulated. For example, the block spin of soft magnetic materials is chosen to be much larger than that in hard magnetic materials.

It is crucial to relate the heat generation (core loss) to material parameters (exchange coupling constant [J], anisotropy constant [K_1], and microstructure, such as grain size and particle size). The wall energy γ in equation (4) is related to $(JK_1)^{1/2}$, and the restoring force will include the dipolar term.

For nanoparticles where only a single domain exists, the coherent rotation is believed to be due to the reversal mechanism [23, 24, 87]. However, recent theory has suggested that even in very small particles, edge domains exist [86]; in most experiments, domains always nucleate because of impurities, dislocations, and surface boundary effects. In MB samples, the impurities and dislocations are expected to be high, and magnetization reversal is almost certainly due to domain wall motion. The theoretical approach presented for the dynamics of magnetic domain wall motion is thus applicable to our study.

For nanocrystalline particles, domain walls can easily nucleate and move rapidly because of a much smaller effective anisotropy K_1. Therefore, significant heat generation is expected from these nanocrystalline particles. The effective anisotropy and dipolar interaction can be calculated using a random anisotropy model that was originally developed by Cullan, Yu and Cullen [88] and Alben, Becker, and Chui [89] for amorphous materials. The basic idea, sketched in Figure 3, starts from an assembly of ferromagnetically coupled grains of size D with magnetocrystalline anisotropies of K_1 oriented at random. The effective anisotropy affecting the magnetization process results from an average over the $N = (L_{ex}/D)^3$ grains with the volume

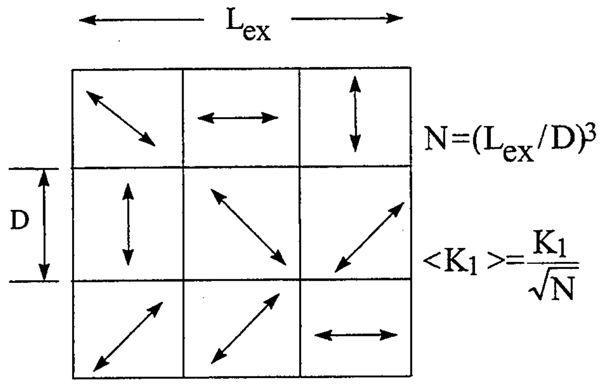

Figure 3. Schematic Representation of the Random Anisotropy Model.

$V = L_{ex}^3$, where $L_{ex} = (A/K_1)^{1/2}$ is the ferromagnetic exchange length and A the exchange stiffness. For a finite number (N) of grains, there will always be a preferred direction determined by statistical fluctuations. Consequently, the resulting anisotropy density $< K_1 >$ is determined by the mean fluctuation amplitude of the anisotropy energy of the *N* grains (i.e., $< K_1 > \approx K_1/\sqrt{N}$). This random anisotropy model was applied to explain the behavior of nanocrystalline materials by Herzer [90, 91] using a dimensionality-like argument. The average magnetocrystalline anisotropy, $< K_1 >$, is predicted to scale with the structural correlation length D as $< K_1 > \propto K_1(D/L_{ex})^6$, and coercive field and permeability is thus scaled with D^6. Experimental data shows this correlation (Figure 4). The dipolar term (g), which scales with grain size as $g_0(L_{ex}/D)^3$, can also be included in simulations.

The frequency spectrum of nanocrystalline particles and nanoparticles can be simulated as a function of external field, frequency, anisotropy constant, and particle and grain size. In particular, the peak in the complex component of permeability, which signifies domain wall resonance, can be investigated as a function of these parameters. The information on particle and grain size can be used to design the structure, and material composition can be selected based on exchange and anisotropy constants. The induction system parameters, such as field and frequency, will be optimized to maximize heating. The core loss and permeability spectrum of the designed material can be measured using a high-frequency hysteresis loop tracer and an impedance analyzer.

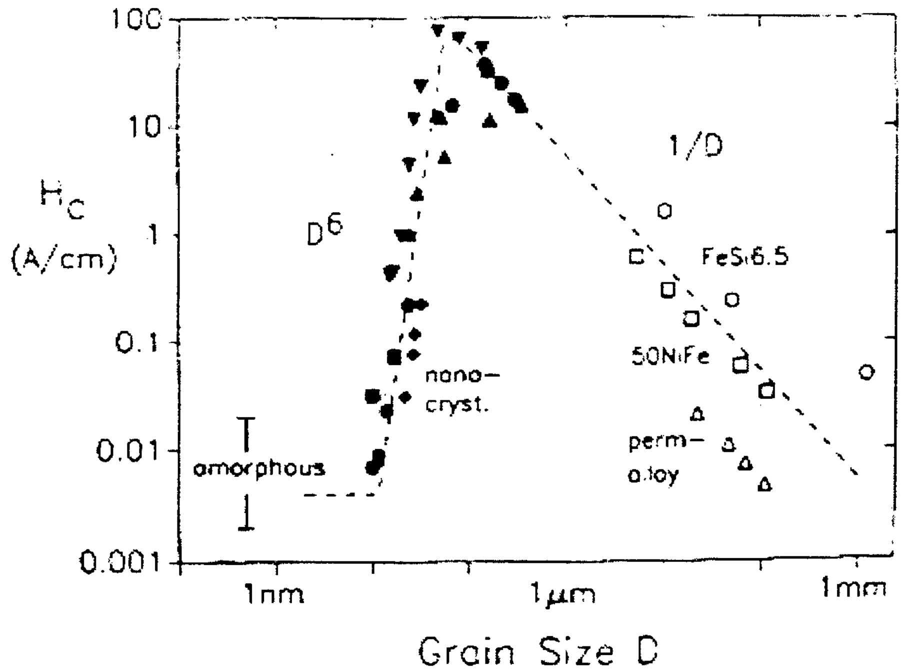

Figure 4. Coercivity vs. Grain Size D for Various Soft Magnetic Materials: Fe-Nb-Si-B (▲), Fe-Cu-Nb-Si-B (●), Fe-Cu-V-Si-B (◆), Fe-Zr-B (■), Fe-Co-Zr (▼), FeNi-Alloys (Δ and □), and FeSi 6.5 weight-percent (○).

2.4 Ferromagnetic to Paramagnetic Phase Transition and Superparamagnetic Relaxation. The second-order magnetic transition from the ferromagnetic to the paramagnetic phase is characterized by the T_c, above which no energy will be absorbed from the external magnetic field and thus no heat will be generated. When nanoscale magnetic particles are embedded in an insulating matrix, negligible heat will be generated from eddy currents. Therefore, the T_c can be used as a smart temperature controller, above which heat generation will automatically shut off.

The T_c can be varied by alloying different materials or using different particle sizes. The latter has not been studied, mainly due to superparamagnetic relaxation. For a single domain particle, when thermal energy is high enough to overcome the energy barrier that restricts the spins from switching direction, the magnetic moments within a particle can rotate rapidly in unison [92–94]. This behavior resembles the paramagnetic phase, except that the particle carries

a giant moment (sum of individual atomic moments in the whole particle), hence the term "superparamagnetism." The susceptibility (χ) in a paramagnetic phase can be written as

$$\chi\,(\mathrm{emu/V}) = \frac{N(p_{eff}\mu_B)^2}{3k_B(T-q)}, \tag{3}$$

where N is the number of paramagnetic moments of an effective atomic moment $p_{eff}\,\mu_B$, and k_B is the Boltzmann constant. In the case of a superparamagnet, N is the number of magnetic particles per unit volume and $p_{eff}\,\mu_B$ is the magnetization of the magnetic particles, which is many times higher than the atomic moment. Clearly, the slopes associated with two phases in the $1/\chi$ vs. T curve will be different, and the T_c can thus be determined. It should be pointed out that the particle size distribution may complicate the analysis. However, since the moments associated with the two phases are very different, the determination of T_c is possible. As an independent check, T_c can also be measured using DSC [95] and by performing specific-heat measurements.

It is important to determine whether the superparamagnetism will affect induction heating, which is typically carried out in the kHz–MHz frequency range. The simplest form of the superparamagnetic relaxation time (τ) can be described by the Arrhenius relation [87]

$$\tau = \tau_0 \exp(KV/k_B T), \tag{4}$$

where τ_0 is the characterization time and KV is the total anisotropy energy (energy barrier) including magnetocrystalline, shape, and magnetoelastic anisotropies. In the case of iron and $Fe_{50}Ni_{50}$, large values of K, in excess of 10^7 erg/cm^3, have been observed [96–98]. This enhancement of K in part accounts for the very large coercivities observed in magnetic particles [99], which can significantly increase the hysteresis loss and heat generation. The value of τ_0 was estimated to be 10^{-11} s from neutron diffraction data [100] and 10^{-13} s from Superconducting Quantum Interference Device (SQUID) magnetometer and Mössbauer spectroscopy [97].

Therefore, considering a magnetic particle of 10 nm at $T = 300$ K, the relaxation rate ($1/\tau$) is much smaller than the frequency of interest (0.1 to 100 MHz). Therefore, these moment rotations have no impact on induction heating.

Although the dimensional dependence of T_c in nanoparticles has not been studied, it has been found that finite-size scaling [101] can be used to explain phase transition behavior in reduced dimension structures. This theory predicts that the shift in the transition temperature from that of the bulk should depend on a dimension of the system in the following manner:

$$\frac{T_c(d) - T_c(\infty)}{T_c(\infty)} = \left(d/d_0\right)^{-\lambda}, \tag{5}$$

where d is the sample dimension, and d_0 should be of the order of the characteristic microscope dimension of the system, such as the lattice parameter. The exponent λ is predicted to be related to the correlation length exponent by $\lambda=1/\nu$.

This behavior has been widely observed in the T_c of multilayer ferromagnetic systems [102] and spin freezing temperature (T_g) in spin-glass systems [103, 104]. The spin freezing temperature in granular materials has also been found to follow the finite-size scaling rule [26]. The only study on T_c of nanoparticles was done using ferrites [105], where the value of T_c was found to increase with decreasing particle size. This observation was later argued to be due to the less-random cation distribution for large particles and not to finite size effects [106, 107].

An alternative and more reliable way to control the T_c is to use alloys. A wide selection of materials is available (e.g., Fe_xCr_{100-x} [$0 < x < 70$, 770 °C $> T_c > 0$ °C], Ni_xCr_{100-x} [$0 < x < 8$, 370 °C $> T_c > 0$ °C], and Ni_xCu_{100-x} [$0 < x < 40$, 370 °C $> T_c > 0$ °C]). These alloys also have excellent corrosion and oxidation resistance. The disadvantage is the low magnetization compared to pure magnetic elements, which may reduce the heat generation capability. A study of T_c in magnetic nanoparticles should address the issues of dimensional dependence and alloy

composition and enable an investigation into the scaling relation in the zero dimension. To study the true T_c, particles can be embedded in a polymer or ceramic matrix to eliminate interparticle interactions.

3. Potential Applications

A number of fundamental issues in nanoparticle design—particle size, frequency, stoichiometry, and T_c—are common to the two applications that will be explored in this research program. The science base for design and synthesis of nanoparticles for hysteresis heating that will be developed as part of the proposed effort will provide a starting point for identifying nanoparticle systems that can be used in the two proposed applications.

3.1 Polymer Processing. Electromagnetic induction is a well-known and widely used heating technique for metals and alloys. More recently, significant research [108–116] has been undertaken to adapt induction-heating techniques to polymers for benefits such as reduced cost and processing times. Heat generation, in a material subjected to an alternating magnetic field, occurs due to joule-type losses [109, 112, 115] (using metal mesh susceptors) or hysteresis [117–119] (using magnetic particles). Both techniques can result in very rapid localized heating, which is ideal for polymer processing.

Existing polymer processing, bonding, and repair techniques require autoclaves, which have size restrictions and are quite costly. Rapid, noncontact, and localized heating will allow for accelerated cure of thermosetting polymers or the rapid interdiffusion of thermoplastic polymers by concentrating energy at the interphase of interest. The design and controlled multiaxis motion of the induction coil can also cure complex polymer composite parts. The advantages of induction-assisted processing include reduced cost and processing time per part, rapid manufacturing, and the potential for increased design complexity.

Of the two heat-generation techniques, hysteresis heating is the focus of this report. A significant advantage to this technique over joule-heating techniques is using the T_c of the

ferromagnetic material as a means of automatic or smart temperature control. By choosing materials such that T_c is within the processing temperature window of the polymer, one can automatically control the process temperature. However, one must also ensure that hysteresis heating is the sole heat-generation mechanism (no joule losses), and this can be achieved by using well-dispersed particles in the polymer. An example of this concept is shown in Figure 5. The nickel/polysulphone susceptor shows very rapid rise to steady-state temperature (~350 °C) once the induction coil is activated and then maintains the temperature regardless of any increase in input power. Polysulphone is a commercially used thermoplastic with a manufacturer-recommended processing window between 300 and 360 °C, and nickel has a T_c of 354 °C, resulting in an ideal combination.

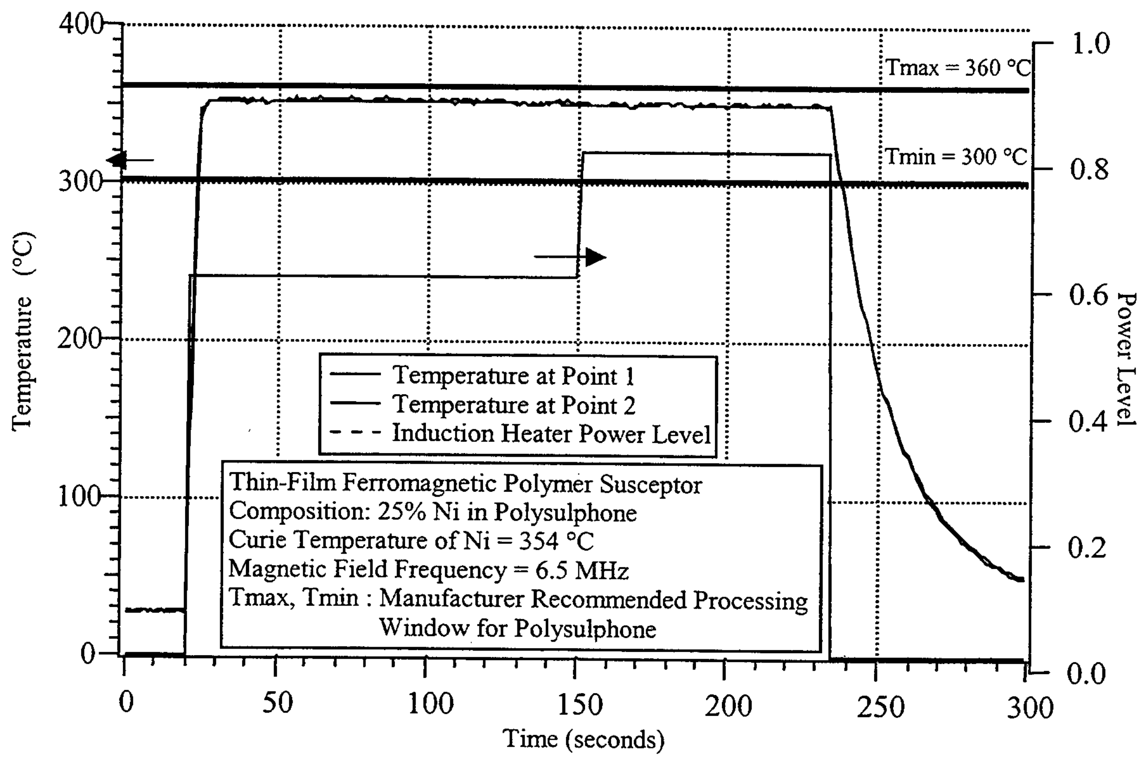

Figure 5. Self-Regulating Temperature Profile for Ferromagnetic Polymer Susceptor. The Two Horizontal Lines at 300 °C and 360 °C Represent the Lower and Upper Bounds on the Process, Respectively.

By choosing ferromagnetic particle systems with T_c between 100 and 400 °C, it is possible to process any thermoset or thermoplastic with a built-in smart temperature control capability. In addition, by using the nanoparticles as additive fillers in the polymer, one can easily adapt existing processing techniques for hysteresis-heating-based processing of composites, with minimal loss in mechanical performance.

The science base for designing and synthesizing nanoparticles for hysteresis heating can provide a starting point in identifying ferromagnetic nanoparticle systems that can be used for polymer processing. Thermal requirements or cure cycles for the processing of polymers should be used to determine the optimal nanoparticle system, particle parameters and induction power, and frequency requirements. Ideally, it is desired that the power input, frequency and particle loading be as low as possible for sufficient heating to completely process the polymer.

Initial tests may focus on heat-transfer dynamics in nanoparticle/polymer systems. A calorimeter-type setup could be built into a hysteresis loop tracer to measure heat generation and correlated with ac hysteresis measurements of the particle system. Nanoparticles could be mixed with polymers for test samples, with particle size, particle composition, volume fraction, magnetic field, and frequency being the parameters. Particles could be mixed with polymers by either solvent casting techniques (e.g., dimethylacetamide and polysulphone) or milling particles with polymer powders and compression molding (e.g., milled nickel particles in polysulphone powder, molded at 350 °C). In both techniques, oxidation of nanoparticles could be minimized by reducing the exposure to air or using surface-treated particles. Effects of exposure to controlled environments (oxygen and air) on particle heating characteristics should also be quantified. Nanoparticle distribution in the polymer could be examined and quantified using SEM and TEM and then used as input for heat transfer simulations. Key results guiding the design of nanoparticles include actual polymer temperature, as compared to particle T_c, automatic temperature control capability, and heat generation.

Based on a finite element model for heat-transfer simulations in particle/polymer systems, nanoparticle systems that meet the thermal requirements for polymer processing could be

identified to ensure that particles are viable and optimized. Thin films could be fabricated using the polymer and nanoparticle system by solvent casting or compression molding and used in the polymer bonding and cure demonstrations. A new concept for curing polymers and composites using hybrid films consisting of different size particles could also be investigated. Typical polymer cure cycles involve multiple temperature steps and dwell times. For example, a typical 350 °F (177 °C) thermoset (TS) may be cured by heating to 250 °F (121 °C), dwelling for 1 hr, and then heating to 350 °F (177 °C) and dwelling for 2 hr. The same steps can be duplicated using a two-material hybrid nanoparticle system and induction heating, with one material having T_c near 250 °F (121 °C) and the other near 350 °F (177 °C) and different optimal frequencies (~700 kHz and ~4–5 MHz) for maximum heating.

3.2 Selective Cell Death. The biological component described in this report is designed to directly test the ability of intracellular nanoparticles to cause selective cell death. These experiments would examine how varying parameters such as particle size, composition, and density, as well as induction frequency and power level, can be used to optimize hyperthermic damage. Hyperthermia is generally thought to cause immediate cell damage and necrosis through protein denaturing and loss of membrane integrity. However, more recent work has also demonstrated effects of moderate (43–46 °C) hyperthermia on DNA damage and apoptosis in various cells, both directly [120–123] and with lag periods requiring restoration of normal incubation temperatures [124]. Therefore, these experiments should examine both primary necrosis and apoptosis at varying times after hyperthermic treatment. Success in selective necrosis and apoptosis would allow the development of nanoparticle-based alternative treatments for cancer.

The general approach is to microinject magnetic nanoparticle suspensions of varying composition into individual cells, subject these cells to inductive heating of varying degree and time, and assay for cell death. Initial experiments could be performed on uninjected cells subjected to passive heating in a DSC to determine the sensitivity and onset of cell death or necrosis and apoptosis to such variables as rate of temperature change, maximum temperature exposure, and length of exposure. Optimal values of temperature rates, maximum temperature,

and duration of exposure would dictate the choice of magnetic nanoparticles and parameters for induction-heating experiments.

3.2.1 Experimental Cell Bioassays. Two types of cell systems are commonly used to study nanoparticle heating properties. The first is the amphibian (*Xenopus*) oocyte model, widely used in microinjection protocols for ribo-nucleic acid (RNA) expression studies or in-vitro fertilization studies [125–127]. To isolate individual oocytes, ovarian lobes are dissected from anesthetized frogs. A total of 10 to 50 stage V–VI oocytes are then removed and manually defolliculated by blunt dissection [125, 128]. Each cell is placed in a well, formed from the mesh of a piece of Nitex filter placed in a Petri dish. The cells are immersed in a modified Barth's solution (MBS). The oocytes can then be rapidly microinjected under a stereo microscope using a micromanipulator and an injection system (PicoPump, WPI, Inc.). Micropipettes are prepared from 1-mm glass capillaries with internal fiber, pulled on a Kopf pipette puller, and beveled to a final tip diameter of 5–10 μM. Generally, injected volumes are 10–50 nl (2–10% of cell volume). Injected cells are then transferred to individual wells of a 96 well culture plate, containing MBS with 0.2% Trypan blue dye to screen for membrane integrity (dye exclusion assay). This model system allows for rapid screening of nanoparticle properties at the individual cell level.

The second assay system consists of cell cultures in various configurations. Both continuous cell lines, including 3T3 fibroblasts and MDCK kidney epithelial cells, and primary cell cultures of rat liver would be assayed. Cells lines are maintained at 37 °C in appropriate growth media (DMEM, RPMI 1640) with fetal bovine serum and subcultured at intervals of 7–14 days. For experiments, cells are plated onto 35-mm collagen-coated plastic Petri dishes. Rat hepatocyte cultures are generated using a collagenase dissociation method [129] and cultured on an artificial extracellular matrix (Matrigel, Becton Dickinson). Both the continuous and primary cell lines could be used in either subconfluent or confluent configurations to assess collateral damage at varying distances from the microinjected target cell. Single cells are microinjected as previously described, except using an inverted, compound microscope with phase contrast optics, micropipets with smaller tip diameters of approximately 1 μm, and reduced injection volumes.

Following injection, the culture medium is replaced with physiological salt solution containing 0.2% Trypan blue. Induction-heating and cell death assays would be performed on the entire monolayer. Controls would include both oocytes and cultured cells injected with carrier solution alone, as well as nanoparticle-injected cells not subjected to the induction heating. The latter would provide information about the cytotoxicity of nanoparticles.

3.2.2 Heating Tests. Individual microinjected oocytes or cultured monolayers could be subjected to heating protocols of varying intensity and duration. Initial tests would use a DSC for passive heating under controlled conditions, with known heating rate, maximum temperature, and duration of heating. These tests will help determine the ideal heating conditions for cell death. Heating rates and maximum temperature would determine the composition, particle size, and T_c of the magnetic nanoparticle system, as well as the power and frequency of the induction heating system.

Individual cell and cell cultures would be microinjected with the selected nanoparticle system and inductively heated according to the heating conditions determined by DSC tests; cell death could then be assessed. It is expected that the induction-heating tests would duplicate the DSC test results and demonstrate the feasibility of this application. The final step would be to assess collateral damage and investigate means to mitigate it; this could be done by injecting selected cells with magnetic nanoparticles and assessing damage to surrounding cells after induction heating.

3.2.3 Cell Death Assays. Immediately after the thermal challenge, cells are examined under light microscopy for Trypan blue uptake. Positive staining indicates loss of membrane integrity and necrosis. Negative cells are then transferred back to an incubator set at normal growth temperature (37 °C) and monitored for an additional, variable period of 1–6 hr. At varying times during this period (1–6 hr post-treatment), some cells would also be assayed for apoptosis. The assay system is based on an in-situ fluorescence variation of the TUNEL (TdT-mediated dUTP-X nick end labeling) method (In Situ Cell Death Detection Kit, Boehringer Mannheim). Briefly, this method allows direct fluorescence visualization of apoptotic cells, based on the predictable

DNA fragmentation that accompanies apoptosis and the use of the enzyme terminal deoxynucleotidyl transferase to attach fluorescent derivatives to free 3'-OH groups on the DNA strands. This approach would allow for rapid screening of apoptosis in individual microinjected oocytes, as well as target and collateral cell death in cultured monolayers.

4. Summary

The goal of this multidisciplinary research would be to establish the science base for the design and synthesis of nanoparticles for hysteresis heating, with potential applications ranging from novel polymer processing techniques to alternative cancer treatments. This research plan would focus on *maximizing* hysteresis losses in magnetic particle systems, while research to date has focused on *minimizing* hysteresis.

4.1 Particle Design. The following are the primary barriers that need to be addressed.

- Magnetization dynamics in high-frequency magnetic fields.
- Effects of magnetic phase transition on hysteresis heating (energy loss).
- Identification of particle composition, size, microstructure, induction power, and frequency for maximum heat generation or power loss at a specified Curie temperature.

The research described in this report would establish the science base for design and synthesis of nanoparticles for hysteresis heating applications.

4.2 Polymer Processing. Magnetic particle systems with T_c between 100 and 400 °C would be chosen for polymer processing demonstrations. Heat-transfer simulations and experiments of nanoparticle/polymer systems will be performed to ensure that particles are viable and optimized for hysteresis heating applications.

4.3 Selective Cell Death. The biological component is designed to explore the ability of intracellular nanoparticles to cause selective cell death. These experiments would examine how varying parameters such as particle size, composition, and density, as well as induction frequency and power level, can be used to cause selective cell death with minimal collateral damage.

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13. ABSTRACT*(Maximum 200 words)*

This report presents the concept for a multidisciplinary research program aimed at establishing the science base for the design and synthesis of magnetic nanoparticles for hysteresis heating, with potential applications ranging from novel composites processing techniques to alternative cancer treatments. Magnetic materials are used in a wide range of applications and designed for maximum efficiency or minimized hysteresis loss. The uniqueness of this project is that, while considerable work has been aimed at reducing hysteresis losses, the converse effect (i.e., increasing hysteresis losses and therefore heat generation) has not been fully studied. Hysteresis-based heating has several advantages over conventional heating techniques, including the fact that it is a very rapid and noncontact process. In addition, the Curie temperature of magnetic materials can also be used as a means of "smart" thermal control. Exploratory basic research through ongoing programs at the U.S. Army Research Laboratory (ARL) has established the feasibility of Curie temperature control and demonstrated the effects of particle size, frequency, and stoichiometry on hysteresis losses. The following scientific barriers are addressed in this report: magnetization dynamics in high-frequency magnetic fields, the effects of magnetic phase transition on hysteresis heating, and the dimensional dependence of Curie temperature in nanoparticles. If carried out, the research program outlined in this report would establish the science base for the design and synthesis of nanoparticles for hysteresis heating applications.

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